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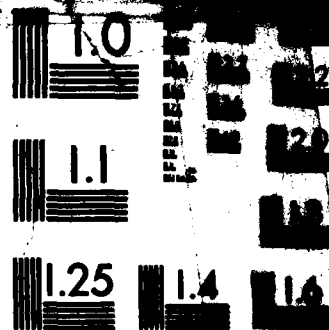
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# NORTHWESTERN UNIVERSITY

## DEPARTMENT OF MATERIALS SCIENCE

TECHNICAL REPORT # 18

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ERRORS DUE TO COUNTING STATISTICS IN THE TRIAXIAL STRAIN  
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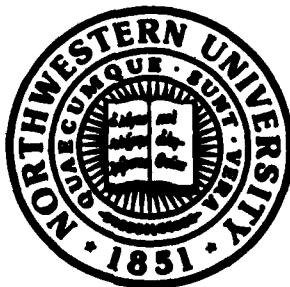
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# ERRORS DUE TO COUNTING STATISTICS IN THE TRIAXIAL STRAIN (STRESS) TENSOR DETERMINED BY DIFFRACTION

P. Rudnik and J. B. Cohen

Department of Materials Science and Engineering  
The Technological Institute  
Northwestern University  
Evanston, IL 60201

## INTRODUCTION

→ Knowledge of the errors in a diffraction measurement of residual strains and stresses is useful information, not only in its own right, but also because it permits automation of a measurement to an operator specified precision.<sup>1</sup> There are three sources of these errors:

(1) Instrumental effects; primarily due to sample displacement, separation of the  $\theta$  and  $2\theta$  axes of the diffractometer, and beam divergence. All three can be estimated<sup>2</sup>, or minimized by employing parallel beam geometry.<sup>3</sup>

(2) Uncertainties in x-ray elastic constants; which can now be evaluated.<sup>4</sup>

and (3) Errors in the diffraction peak position related to counting statistics. (to #173) Equations to evaluate this source have been developed in Ref. 1 for the case of a stress state for which all  $\sigma_{ij}$  ( $i = 1, 2, 3$ ) = 0, with the direction "3" normal to the sample surface, see Fig. 1. This means that the stresses lie only in the surface, e.g., a biaxial stress state  $\begin{vmatrix} \sigma_{11} & \sigma_{12} \\ \sigma_{12} & \sigma_{22} \end{vmatrix}$ . There are, however,

numerous situations when the normal components are appreciable in an x-ray measurement<sup>5,6</sup> and this is generally the case for neutron diffraction because with neutrons the beam can sample a sizeable volume, at a significant depth below the surface<sup>7</sup>. It

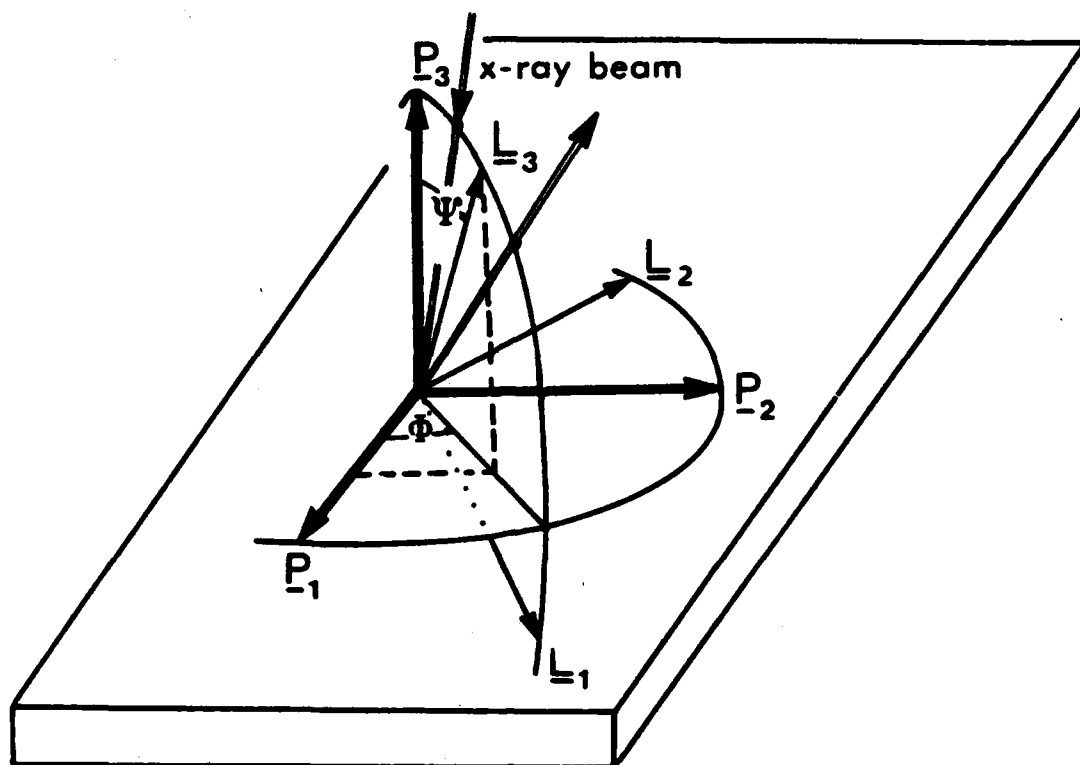


FIG. 1: The axial system. Strains are measured with diffraction by measuring the change in spacing of planes normal to the  $L_3$  direction. (The axes  $P_1$  define the specimen surface.)

TABLE I: STRESS TENSORS ( AND STANDARD DEVIATIONS)  
FOR SPECIMEN C3, REF. 5

DATA SET 1					
537.62	(161.94)	-24.03	(78.81)	-39.15	(4.58)
		550.04	(161.66)	2.31	(3.56)
				78.29	(130.57)
DATA SET 2					
520.60	(137.25)	-4.03	(66.60)	-34.17	(3.21)
		555.19	(137.03)	0.11	(2.69)
				82.20	(110.67)
DATA SET 3					
535.03	(158.28)	-20.13	(77.06)	-40.19	(5.72)
		555.98	(157.99)	-0.98	(4.56)
				86.66	(127.69)
DATA SET 4					
538.53	(146.23)	-30.63	(70.95)	-38.03	(3.83)
		565.37	(146.14)	0.76	(3.89)
				88.18	(117.92)
AVERAGE					
532.95	-19.69	-37.89			
	556.65	0.55			
		83.83			
REFERENCE 5					
		541	-20	-38	
			565	1	
				86	

\* values given in MPa;  $V(d_0)^{1/2} = 0.00016 \text{ \AA}$

is the purpose of this paper to derive equations to evaluate the counting statistical error for the entire three dimensional strain (or stress) tensor,

$$\begin{vmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{12} & \sigma_{22} & \sigma_{23} \\ \sigma_{13} & \sigma_{23} & \sigma_{33} \end{vmatrix}.$$

### BASIC EQUATIONS

We begin with the general equation for the strains ( $\epsilon_{ij}$ ) and how these affect the interplanar spacing "d". (Refer to Fig. 1 for the axial system.) The measurement is made in the  $\theta$  direction, with a sample tilted  $\phi$  from the normal position (which is with the surface normal bisecting incident and scattered beams). Primed quantities refer to strains in the  $L_1$  co-ordinate system, unprimed terms are in the  $P_1$  system.

$$\begin{aligned} \langle \epsilon'_{33} \rangle_{\theta\phi} &= (d_{\theta\phi} - d_0)/d_0 = [\langle \epsilon_{11} \rangle \cos^2 \theta + \langle \epsilon_{22} \rangle \sin^2 \theta + \langle \epsilon_{12} \rangle \sin 2\theta \\ &\quad - \langle \epsilon_{33} \rangle \sin^2 \phi + \langle \epsilon_{31} \rangle + [\langle \epsilon_{11} \rangle \cos \theta + \langle \epsilon_{22} \rangle \sin \theta] \sin 2\phi] \sin^2 \phi \end{aligned} \quad (1)$$

Note that the stress-free spacing,  $d_0$ , is involved. While this term can be eliminated for a biaxial stress state, this is not possible for a general strain or stress tensor, and the reader may consult Ref. 8 for a discussion of problems associated with the measurement of this quantity. When  $\epsilon_{12}$ , or  $\epsilon_{11}$ ,  $\neq 0$ ,  $\epsilon'_{33}$  is not linear with  $\sin^2 \phi$  and has different curvature for  $+\phi$  and  $-\phi$ . The carats imply that the strain values are averaged over the depth of penetration of the incident x-ray (neutron) beam and this is to be understood in what follows, as this additional notation is eliminated below.

Next, we define terms which involve measurements of  $d_{\theta,\phi}$  at plus and minus  $\phi$  tilts of the surface normal.<sup>5</sup>

$$\begin{aligned} a_1 &\equiv 1/2[\epsilon'_{\theta\phi+} + \epsilon'_{\theta\phi-}] = (d_{\theta\phi+} + d_{\theta\phi-})/2d_0 - 1 \\ &= \epsilon_{33} + [\epsilon_{11} \cos^2 \theta + \epsilon_{22} \sin^2 \theta + \epsilon_{12} \sin 2\theta - \epsilon_{33}] \sin^2 \phi \end{aligned} \quad (2a)$$

Clearly,  $a_1$ , should be linear with  $\sin^2 \phi$  and  $\epsilon_{33}$  is the intercept, regardless of  $\theta$ .

$$\begin{aligned} a_2 &\equiv 1/2[\epsilon'_{\theta\phi+} - \epsilon'_{\theta\phi-}] = (d_{\theta\phi+} - d_{\theta\phi-})/2d_0 \\ &= [\epsilon_{11} \cos \theta + \epsilon_{22} \sin \theta] \sin |2\phi|. \end{aligned} \quad (2b)$$

Therefore,  $a_2$  is linear vs.  $\sin|2\phi|$ .

Let:

$$L_1 = da_1/d\sin^2\phi, \quad (3a)$$

$$L_2 = da_2/d\sin|2\phi| \quad (3b)$$

$$\text{Then, at: } \phi = 0^\circ, \quad {}_0L_1 = \epsilon_{11} - \epsilon_{33},$$

$$\phi = 90^\circ, \quad {}_{90}L_1 = \epsilon_{22} - \epsilon_{33},$$

$$\begin{aligned} \phi = 45^\circ, \quad {}_{45}L_1 &= 1/2(\epsilon_{11} + \epsilon_{22}) + \epsilon_{12} - \epsilon_{33}, \\ &= \epsilon_{12} + 1/2({}_0L_1 + {}_{90}L_1). \end{aligned} \quad (3c)$$

and similarly:

$$\text{at } \phi = 0^\circ: \quad {}_0L_2 = \epsilon_{13},$$

$$\text{at } \phi = 90^\circ: \quad {}_{90}L_2 = \epsilon_{23}. \quad (3d)$$

Knowledge of the strain tensor permits the calculation of the stress components ( $\sigma_{ij}$ ) from:

$$\begin{aligned} \sigma_{ij} &= [1/2S_2(hkl)]^{-1} \{ \epsilon_{ij} - \delta_{ij} \{ S_1(hkl)/[3S_1(hkl) \\ &\quad + 1/2S_2(hkl)] \} \cdot [\epsilon_{11} + \epsilon_{22} + \epsilon_{33}] \}. \end{aligned} \quad (4)$$

Here,  $\delta_{ij}$  is the Kronecker delta function and  $S_1$  and  $1/2S_2$  are the x-ray elastic constants which depend on the indices of the diffraction peak,  $hkl$ . (For an isotropic solid these values are  $-v/E$  and  $(1+v)/E$  respectively.)

#### VARIANCES DUE TO COUNTING STATISTICS

For a function  $X = f(x_1, x_2, x_3 \dots)$ , assuming the  $x_n$  are independent, the variance ( $V$ ) is<sup>9</sup>:

$$V(X) = \left(\frac{dX}{dx_1}\right)^2 V(x_1) + \left(\frac{dX}{dx_2}\right)^2 V(x_2) + \left(\frac{dX}{dx_3}\right)^2 V(x_3) + \dots \quad (5)$$

For the straight line,  $y = mx + b$ , the slope and intercept is given by:

$$m = \frac{\sum_1 (x_1 - \bar{x})(y_1 - \bar{y})}{\sum_1 (x_1 - \bar{x})^2} \quad (6a)$$

$$b = (\sum y_1 - m \sum x_1)/N \quad (6b)$$

where  $N$  is the number of data points.

Employing Eq. (5):

$$V(m) = \left[ \frac{\sum_1 (y_1 - \bar{y})}{\sum_1 (x_1 - \bar{x})^2} \right]^2 V(x) + \left[ \frac{\sum_1 (x_1 - \bar{x})}{\sum_1 (x_1 - \bar{x})^2} \right]^2 V(y) \quad (6c)$$

Therefore:

$$V(b) = \frac{\sum_1 (x_1 - \bar{x})^2}{N} \cdot V(m) = \frac{\sum_1 (x_1 - \bar{x})^2}{N} \cdot \left\{ \frac{\sum_1 (y_1 - \bar{y})^2}{\sum_1 (x_1 - \bar{x})^2} V(x) + \left[ \frac{\sum_1 (x_1 - \bar{x})}{\sum_1 (x_1 - \bar{x})^2} \right]^2 V(y) \right\} \quad (6d)$$

Therefore; in terms of  $a_1$  vs.  $\sin^2 \phi$ :

$$V(a_1) = \left[ \frac{\sum_1 (a_{11} - \bar{a}_1)}{\sum_1 (\sin^2 \phi_1 - \overline{\sin^2 \phi})} \right]^2 V(\sin^2 \phi) + \left[ \frac{\sum_1 (\sin^2 \phi_1 - \overline{\sin^2 \phi})}{\sum_1 (\sin^2 \phi_1 - \overline{\sin^2 \phi})^2} \right]^2 V(a_1) \quad (7)$$

The variance in  $\phi$  is negligible, so the first term can be ignored.

Also, from Eq. (2a):

$$V(a_1) = \left[ \frac{da_1}{d(d_{\phi\phi+})} \right]^2 V(d_{\phi\phi+}) + \left[ \frac{da_1}{d(d_{\phi\phi-})} \right]^2 V(d_{\phi\phi-}) + \left[ \frac{da_1}{d(d_0)} \right]^2 V(d_0) \quad (8)$$

Writing Bragg's law in the form  $d = \frac{\lambda}{2\sin\theta}$ , adopting the convention that  $\theta^+$ ,  $\theta^-$  are the  $\theta$  values (in degrees) for the peaks at  $+\phi$ ,  $-\phi$  respectively, and employing Eq. (5):

$$V(d_{\phi\phi+}) = (\pi/180)^2 (\lambda \cos \theta^+ / 2 \sin^2 \theta^+)^2 V(2\theta^+) / 2 \quad (9)$$

and similarly for  $V(d_{\phi\phi-})$ . Recalling Eq. (2a):

$$\left[ \frac{da_1}{d(d_{\phi\phi+})} \right]^2 = \left[ \frac{da_1}{d(d_{\phi\phi-})} \right]^2 = \frac{1}{4d_0^2}, \quad (10a)$$



$$-\frac{da_1}{d(d_0)} = \frac{[d_{\phi\phi+} + d_{\phi\phi-}]^2}{4d_0^4} = d_+^2/4d_0^4. \quad (10b)$$

Thus, we may rewrite Eq. (7):

$$V(\ell_1) = \left[ \frac{\sum_1 (\sin^2 \phi_1 - \overline{\sin^2 \phi})}{\sum_1 (\sin^2 \phi_1 - \overline{\sin^2 \phi})^2} \right]^2 \frac{1}{4d_0^2} \left\{ \left( \frac{\pi}{180} \right)^2 \frac{\lambda^2}{8} \left[ \left( \frac{\cos \theta^+}{\sin^2 \theta^+} \right)^2 v_1(2\theta^+) \right. \right. \\ \left. \left. + \left( \frac{\cos \theta^-}{\sin^2 \theta^-} \right)^2 v_1(2\theta^-) \right] + (d_+^2/d_0^2) V(d_0) \right\} \quad (11)$$

In a similar manner for  $a_2$  vs.  $\sin|2\phi|$ , where  $a_2 \equiv (d_{\phi\phi-} - d_{\phi\phi+})/2d_0 = d_-/2d_0$ :

$$V(\ell_2) = \left[ \frac{\sum_1 \sin|2\phi_1| - \overline{\sin|2\phi|}}{\sum_1 (\sin|2\phi_1| - \overline{\sin|2\phi|})^2} \right]^2 \frac{1}{4d_0^2} \left\{ \left( \frac{\pi}{180} \right)^2 \left( \frac{\lambda^2}{8} \right) \left[ \left( \frac{\cos \theta^+}{\sin^2 \theta^+} \right)^2 v_1(2\theta^+) \right. \right. \\ \left. \left. + \left( \frac{\cos \theta^-}{\sin^2 \theta^-} \right)^2 v_1(2\theta^-) \right] + (d_-^2/d_0^2) V(d_0) \right\} \quad (12)$$

We now propagate these values into the strain and stress tensors.

#### THE STRAIN TENSOR

Abbreviating the intercept of  $a_1$  vs.  $\sin^2 \phi$  as  $I$ , then at any  $\phi$ :

$$\epsilon_{33} = I \text{ (of } a_1 \text{ vs. } \sin^2 \phi), \quad (13a)$$

$$V(\epsilon_{33}) = V(\ell_1) + V(I) \quad (13b)$$

$$V(I) = \frac{\sum (\sin^2 \phi_1 - \overline{\sin^2 \phi})^2}{N} \cdot \left[ \frac{\sum (\sin^2 \phi_1 - \overline{\sin^2 \phi})}{\sum (\sin^2 \phi_1 - \overline{\sin^2 \phi})^2} \right]^2 V(a_{11}) \\ = \frac{1}{N} \frac{[\sum (\sin^2 \phi_1 - \overline{\sin^2 \phi})]^2}{\sum (\sin^2 \phi_1 - \overline{\sin^2 \phi})^2} V(a_{11}) \quad (13c)$$

Now, from Eqs. (3c), at  $\phi = 0^\circ$ :

$$\epsilon_{\ell_1} = \epsilon_{11} - \epsilon_{33} = \epsilon_{11} - I, \quad (14)$$

$$V(\epsilon_{\ell_1}) = 2V(\epsilon_{11}) + V(I).$$

Similarly, for  $\phi = 90^\circ$ :

$$\epsilon_{22} = \epsilon_0 \epsilon_1 + I,$$

$$V(\epsilon_{22}) = V(\epsilon_0 \epsilon_1) + V(\epsilon_0 \epsilon_1) + V(I), \quad (15)$$

and for  $\theta = 45^\circ$ :

$$\epsilon_{12} = \epsilon_0 \epsilon_1 + \epsilon_{33} - 0.5 (\epsilon_{11} + \epsilon_{22})$$

$$+ \epsilon_0 \epsilon_1 - 0.5 (\epsilon_0 \epsilon_1 + \epsilon_0 \epsilon_1),$$

$$V(\epsilon_{12}) = V(\epsilon_0 \epsilon_1) + 0.25 [V(\epsilon_0 \epsilon_1) + V(\epsilon_0 \epsilon_1)]. \quad (16)$$

From Eqs. (3d):

$$V(\epsilon_{12}) = V(\epsilon_0 \epsilon_2), \quad (17)$$

$$V(\epsilon_{23}) = V(\epsilon_0 \epsilon_2). \quad (18)$$

### THE STRESS TENSOR

We define  $Q = S_1 / (3S_1 + 1/2S_2)$  (which is  $[-\nu/1-2\nu]$  for an isotropic solid). Then Eq. (4) may be written, for the diagonal stress components, as:

$$\sigma_{ij} = (1/2S_2)^{-1} [(1-Q)\epsilon_{ii} - Q\epsilon_{kk} - Q\epsilon_{jj}]. \quad (19)$$

Here  $i = 1, 2, 3$ ;  $j = 2, 3, 1$ ;  $k = 3, 1, 2$ .

From Eq. (19):

$$V(\sigma_{ii})^{\frac{1}{2}} = (1/2S_2)^{-1} \{ (1-Q)^2 V(\epsilon_{ii}) + Q^2 [V(\epsilon_{kk}) + V(\epsilon_{jj})] \}^{\frac{1}{2}}. \quad (20)$$

Therefore, with Eqs. (13-15):

$$V(\sigma_{11})^{\frac{1}{2}} = (1/2S_2)^{-1} \{ (2-4Q + 4Q^2)V(\epsilon_0 \epsilon_1) + Q^2 V(\epsilon_0 \epsilon_1) + (1-2Q + 3Q^2)V(I) \}^{\frac{1}{2}}, \quad (21)$$

$$V(\sigma_{22})^{\frac{1}{2}} = (1/2S_2)^{-1} \{ (1-2Q + 4Q^2)V(\epsilon_0 \epsilon_1) + (1-2Q + Q^2)V(\epsilon_0 \epsilon_1) + (1-2Q + 3Q^2)V(I) \}^{\frac{1}{2}}, \quad (22)$$

$$V(\sigma_{33})^{\frac{1}{2}} = (1/2S_2)^{-1} \{ (1-2Q + 4Q^2)V(\epsilon_0 \epsilon_1) + Q^2 V(\epsilon_0 \epsilon_1) + (1-2Q + 3Q^2)V(I) \}^{\frac{1}{2}} \quad (23)$$

$$V(\alpha_2)^{\frac{1}{2}} = (1/2S_2)^{-1} [V(\epsilon_1) + 0.25 [V(\epsilon_1) + V(\epsilon_1)]]^{\frac{1}{2}}, \quad (24)$$

$$V(\alpha_1)^{\frac{1}{2}} = (1/2S_2)^{-1} V(\epsilon_2)^{\frac{1}{2}}, \quad (25)$$

$$V(\alpha_2) = (1-2S_2)^{-1} V(\epsilon_2). \quad (26)$$

### EXAMPLES

To illustrate the typical magnitudes of the errors due to counting statistics, we employed data from Ref. 5, for a ground steel specimen, that is we used the peak positions and the variances in these positions with Eq. (9). [Formulae to calculate this variance for the parabolic fit employed in Ref. 5 are given in Ref. 1; for other types of fits the appropriate equation may be substituted.] The resultant errors are given in Tables I-III. For the first two tables it was assumed that the error in  $d_{\phi}$  was the actual measured value. If there is no preferred orientation, the intensity of the peak changes little with the  $\phi$ -tilt. In this case, Tables I and II show the effect of the uncertainty in  $d_0$ ; reducing this error all the stress components by the same proportion, except  $\alpha_1$ ,  $\alpha_2$ , which remain relatively unaffected, because the role of the error in  $d_0$  is damped by  $(d_0)^2$  in Eq. (12).

If there is preferred orientation, the peak intensity can vary greatly with  $\phi$  and there will be large variances contributing to  $V(\epsilon_1)$  from weak peaks. This was minimized in the following way. The average variance,  $\sigma_1$ , in the 2 $\theta$  peak position for  $+\phi$  and  $-\phi$  was obtained and the weighting factor  $c_1$  was formed:

$$c_1 = (1/\sigma_1^2) / \sum_i (1/\sigma_i^2) \quad (27)$$

The Eqs. 11 and 12 were then altered to multiply  $V_1(2\theta^+)$ ,  $V_1(2\theta^-)$  terms by this weighting for Table III. There is only a small difference (between Tables II and III) because of the lack of texture in the specimen; the peak intensity changed only by about 8 pct with  $\phi$ . With more severe preferred orientation the effect will be larger.

### CONCLUDING REMARKS

There are now adequate equations for calculating errors in stress

TABLE II: STRESS TENSOR AND STANDARD DEVIATIONS WHEN  
 $V(d_0)^{\frac{1}{2}} = 0.00004 \text{ \AA}^*$

		DATA SET 1			
539.74	(48.24)	-24.03	(24.50)	-39.15	(4.58)
		552.16	(47.26)	2.30	(3.56)
				80.41	(38.96)
		DATA SET 2			
520.60	(40.52)	-4.03	(19.95)	-34.17	(3.21)
		555.19	(39.73)	0.11	(2.69)
				82.20	(32.75)
		DATA SET 3			
535.03	(47.81)	-20.14	(24.35)	-40.19	(5.72)
		555.98	(46.81)	-0.98	(4.56)
				86.66	(38.84)
		DATA SET 4			
538.53	(42.84)	-30.63	(21.10)	-38.03	(3.83)
		565.37	(42.51)	0.76	(3.89)
				88.18	(34.67)

\*values given in MPa

TABLE III: WEIGHTED STRESS TENSOR AND STANDARD DEVIATIONS\*

		DATA SET 1			
536.24	(48.56)	-24.62	(24.56)	-38.33	(4.59)
		554.65	(47.60)	2.90	(3.65)
				80.68	(39.22)
		DATA SET 2			
520.29	(41.84)	-6.04	(20.11)	-34.82	(3.55)
		560.22	(40.60)	1.56	(2.73)
				85.29	(33.76)
		DATA SET 3			
532.56	(48.15)	-7.90	(24.93)	-39.66	(5.81)
		549.83	(47.16)	-2.81	(4.57)
				82.21	(39.16)
		DATA SET 4			
539.23	(42.88)	-31.22	(21.23)	-38.28	(3.85)
		565.17	(42.65)	-0.59	(3.94)
				88.98	(34.68)

\*  $V(d_0)^{\frac{1}{2}} = 0.00004 \text{ \AA}$ ; values given in MPa

measurements due to instrumental effects, counting statistics and in the x-ray elastic constants. We would like to conclude this paper with a plea to the community making stress measurements via diffraction to regularly report these errors with their data. It is all too common for investigators to repeat a measurement (of stress or an elastic constant) once and to use the difference as an error estimate. Another practice is to dust a stress-free powder on the specimen surface and to use a (single) measurement of the stresses measured with this powder as an error estimate. Finally, some report an error in a slope vs.  $\sin^2\psi$  obtained by least-squares, but ignore the uncertainty in each point in this plot in estimating errors. None of these procedures is particularly satisfying in a statistical sense. Of course, if time permits, the average of, say, ten repetitions of a measurement is the best of all error estimates. If this cannot be done, error estimates from the available equations are far more satisfactory than the currently all - too common procedures.

#### ACKNOWLEDGEMENTS

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Evanston, IL 60201

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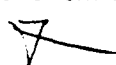
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## 11. SUPPLEMENTARY NOTES

## 12. SPONSORING MILITARY ACTIVITY

Metallurgy Branch  
Office of Naval Research

## 13. ABSTRACT

The errors are derived for the diffraction measurement of the three dimensional stress and strain tensor due to counting statistics. 

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## KEY WORDS

## LINK A

## LINK B

## LINK C

ROLE

WT

ROLE

WT

ROLE

WT

Stresses  
Residual stresses  
Errors in residual stresses  
Counting statistical errors in stresses

**END**

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**9-85**

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